

SYNTHESIS AND CHARACTERIZATION OF CUO NANO PARTICLES USING SIMPLE GLUCOSE REDUCTION METHOD

G. DILEEP KUMAR¹ & N. NATARAJAN²

¹Ph.D. Scholar, Department of Seed Science and Technology, Tamil Nadu Agricultural
University, Coimbatore, India

²Professor, Department of Seed Science and Technology, Horticultural College and Research
Institute for Women, Trichy, India

ABSTRACT

The copper nano particles were synthesized from copper sulfate pentahydrate aqueous solution under the low hydrothermal condition at 80°C. Nanoparticles were synthesized by using a well-controlled surfactant assisted solution phase synthesis. The method involved reduction of copper acetate by glucose in a mixture of water and ethylene glycol serving as the base fluid. The synthesized particles are characterized by X-ray diffraction technique, electron diffraction X-ray analysis transmission electron microscopy (TEM) and scanning electron microscopy (SEM) for phase structure, chemical composition and morphology respectively. The synthesized nanoparticles are confirmed to be copper. It is reported that the method may be feasible for large scale production of nano CuO particles for practical applications.

KEYWORDS: Cuprous Oxide, Nanoparticles, Thermal Conductivity

INTRODUCTION

Metal oxides represent the most diverse class of materials with properties covering almost all aspects of biological science and material science. Copper oxide is used in consumer products such as cosmetic and antimicrobial properties. The thermal conductivity of heat transfer fluids plays an important role in the development of energy-efficient equipment's in the field of microelectronics, transportation, military devices and biomedical industry (Liu et al., 2006; Zhu et al., 2004a). Nanoscale materials are currently of great interest due to their unique electronic, optical, and mechanical properties. Particularly semiconductor nanoparticles have drawn a significant interest in recent years because of their special properties such as a large surface to volume ratio, increased activity, special electronic properties and unique optical properties as compared to those of the bulk materials. The oxides of transition metals are an important class of semiconductors having applications in multiple technical fields like solar energy transformation, magnetic storage media, electronics and catalysis (Wolhfarth *et al.*, 1980; Mallinson, 1987; Bradley *et al.*, 1976; Mitsuyu *et al.*, 1982). Among the oxides of transition metals, copper oxide nanoparticles are of special interest because of their efficiency as nanofluids in heat transfer application (Chang *et al.*, 2011), secondly it is the basis of several high-T_c superconductors (Wang *et al.*, 2002). CuO is a semiconducting material with a narrow band gap and used for photoconductive and photo thermal applications (Rakhshni *et al.*, 1986). Opposite to n-type semiconducting metal oxides, cupric oxide (CuO) is a p-type semiconductor with a band gap of 1.2–1.9 eV. Its applications also include catalysis, lithium–copper oxide electrochemical cells, solar cells, and gas sensors (Zhang *et al.*, 2006; Cruccolini *et al.*, 2004; Kim *et al.*, 2008; Li *et al.*, 2008). The nanoparticles, plates, and nanowires of CuO were also reported to sense NO₂, H₂S and CO

(Cruccolini *et al.*, 2004; Li *et al.*, 2008) up to now, the reports on the preparation and characterization of nanocrystalline CuO are comparatively few to some other transition metal oxides such as zinc oxide (ZnO), titanium dioxide (TiO₂), tin dioxide (SnO₂) and iron oxide (Fe₂O₃) material an interesting candidate to investigate. Copper and copper oxide nanoparticles have received much attention recently due to their promising properties. Synthesis of nanofluids using these nanoparticles ensures a logical move to satiate the ever increasing cooling need of many industrial technologies.

MATERIALS AND METHODS

Preparation of Cuprous Oxide Nanofluids

All the reagents used in the experiment were of analytical grade and used without further purification. The cuprous oxide nanofluids were prepared by the chemical reduction of copper aqueous of copper acetate (20 mL, 0.25 M) was made sufficiently alkaline by addition of ammonium hydroxide. To this deep blue solution, a solution of SLS in ethylene glycol (10 mL, 0.01 M) was added and stirred for 15 min. Glucose (2.5 g) was then added and heated to 75 °C with stirring. When the colour of the solution changed from blue to golden yellow, sulphuric acid was added to neutralize the initially added base and heating was continued for some time. Finally the brown solution was cooled to obtain a cuprous oxide nanofluid. Similar reactions were carried out by varying the concentrations of reactants and also by varying the dilution of the reaction mixture.

Characterization

The prepared cuprous oxide nanofluid were subjected to various characterized techniques like X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning Electron microscope with EDS (SEM/EDS) and particle size analyzer.

Scanning Electron Microscope (SEM)

SEM FEI QUANTA 250 was used to characterize the size and morphology of the nanoparticles. Sample of test nanoparticles (0.5 to 1.0 mg) was dusted on one side of the double sided adhesive carbon conducting tape, and then mounted on the 8mm diameter aluminum stub. Sample surface were observed at different magnification and the images were recorded.

Transmission Electron Microscope (TEM)

TEM FEI TECHNAI SPRIT, make or source was used to analyze the sample. Dilute suspensions of nanoparticles (0.50 mg) in pure ethanol (15 ml) were prepared by ultrasonication. A drop of the suspension placed on 300-mesh lacy carbon coated copper grid, dried and the images were recorded at different magnification.

X-ray diffraction

The X-ray diffractograms have been recorded on Powder XRD (Bruker D8 Advance Powder X-ray Diffractometer, Germany). The machine exploits Cu-K α radiation (0.154 nm) for measuring the crystalline nature of atoms in the material (Toraya, 1986). The diffractograms were recorded in the range of $2\theta = 10-80$ degrees at a scanning speed of 0.080 and step times 1s at room temperature 25°C.

RESULTS AND DISCUSSIONS

SEM and TEM Analysis

The formation of CuO NPs was initially confirmed with change in color of the reaction mixture (Fig. 1) due to surface plasmon resonance phenomenon provides a convenient signature to indicate the formation of CONPs in the reaction mixture (Krithiga *et al.*, 2013). The surface morphology of the nanoparticles synthesized and examined under SEM revealed that CuO nanocrystals organized into spherical (Fig. 2) measuring 280 to 365 nm diameter with higher magnification on individual particle it appears like dandelion. In this study, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ was used to prepare spherical shaped nano CuO at mild reaction temperature. The same morphology was obtained when using $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ as reported by (Anandan *et al.*, 2007).



Figure 1: Color Change During the Phyto-reduction of CuSO_4 into CuO NPs at the Beginning and after 48 h of Reaction

To confirm the results of SEM, the same nanoparticles were characterized under TEM. CuO nanoparticles were diagnosed primarily to be cylindrical in shape as against spherical under SEM but measured 100 nm in conformity (Fig. 3).

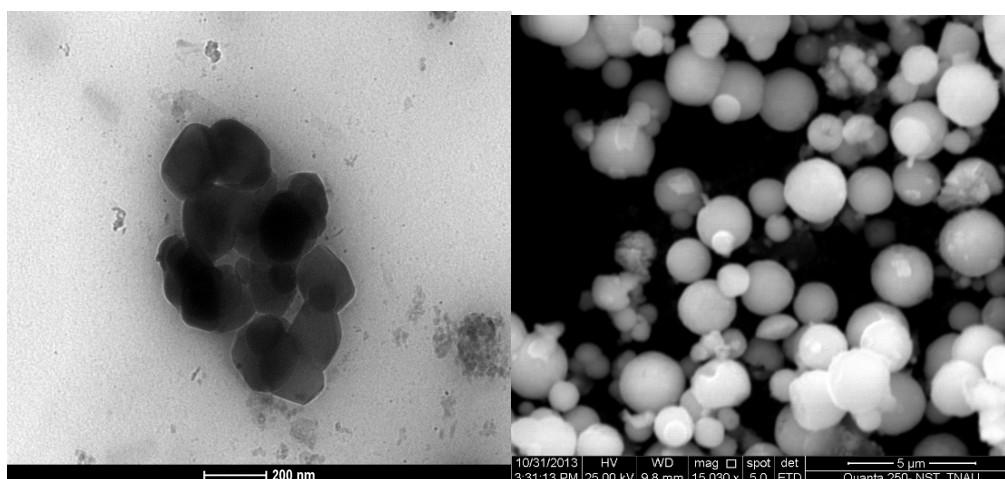


Figure 2: SEM / EDS Image of CuO Nanoparticles Figure 3: TEM Image of CuO Nanoparticles

Powder XRD Analysis

The phase structure and the purity of the CuO were examined by Powder X-ray diffraction studies. The powder XRD pattern of the as obtained cuprous oxide nanoparticles is shown in Figure 4. The peak positions of the sample exhibited monoclinic structure of CuO which was confirmed from the ICDD card No 801916. Further, no other impurity peak was observed in the XRD pattern, showing the single phase sample formation. Lattice parameters of unit cell of CuO is found to be $a = 3.387 \text{ \AA}$. The d-spacing value is in good agreement with the standard values reported by the ICDD Card No 801916.

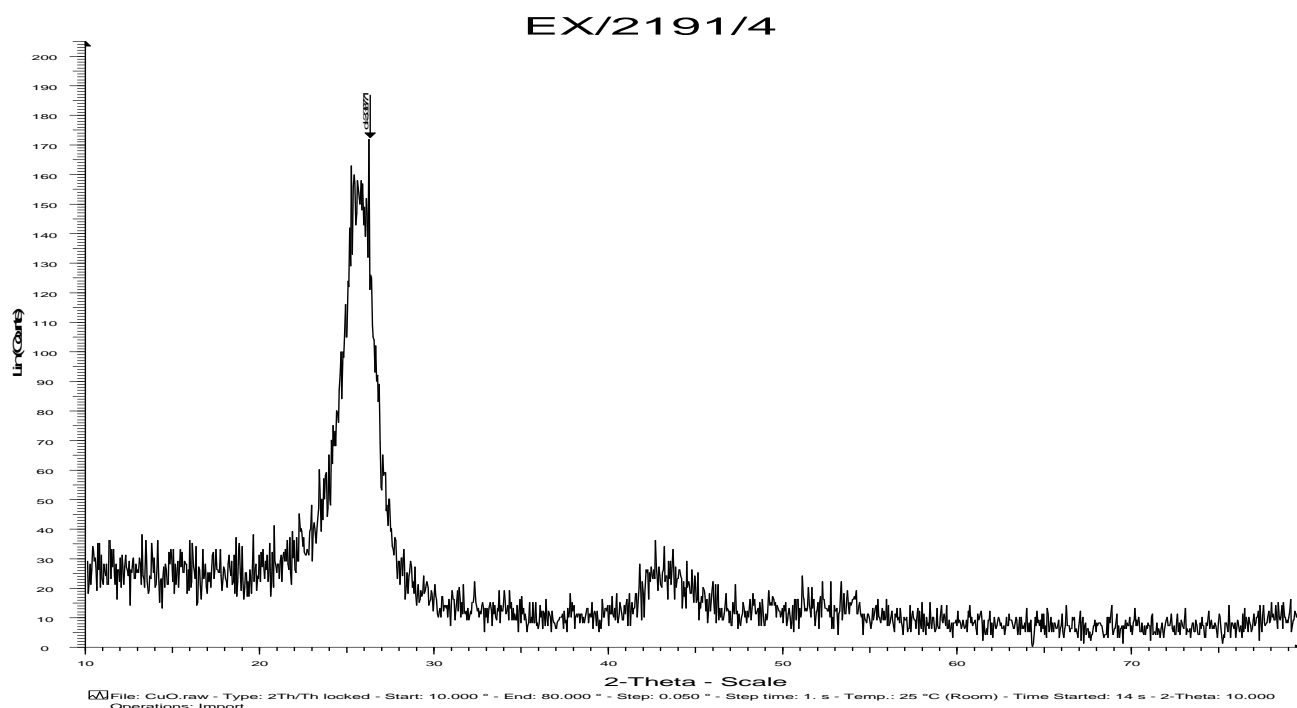


Figure 4: Powder XRD Patterns of CuO Nanoparticles

RESULT OF SURFACTANT

Surfactant concentration also played an important role in controlling the size of the particle as well as stability of the nano particle. The effect of increase in surfactant concentration on size of the particle is shown in Table 1. The results clearly indicate that SLS acts as a capping agent and hence restricts the growth of the particles. As the SLS concentration increases, the particle size decreases.

The presence of surfactant also had an effect on the stability of the fluid. In the absence of surfactant, the particles formed immediately began to settle down, but in the presence of surfactant, this was not the case. Sedimentation measurements showed that the nanofluid prepared in the presence of surfactant was stable up to 3 weeks at room temperature under stationary state. The stability achieved is better than the one by the two-step method wherein they could achieve it for 1 week in case of copper-transformer oil system and 30 h for copper– water system (Xuan and Li 2000). The stability of the fluid could be attributed to the small size of the particles and their uniform distribution.

Table 1: Effect of concentration of SLS on size of copper particles

Concentration of SLS Added (M)	Effective Concentration of SLS in the Reaction Mixture (mM)	Particle Size (nm)
0.00	0	59
0.01	5	38
0.05	25	26
0.1	50	15

CONCLUSIONS

Copper nanoparticles were synthesized by a well-controlled one-step solution phase glucose reduction method. It is confirmed by XRD analysis. The spherical Morphology is confirmed by both SEM / EDS. Here the copper nitrate was reduced to copper and was simultaneously dispersed in the base fluid, in the presence of SLS. The stability period of minimum 4 weeks was achieved in the presence of surfactant. The method is found to be reliable, simple in approach and cost effective.

REFERENCES

- Bradley, F.N. (1976). Materials for Magnetic Functions. Chapter 2. Hayden, New York
- Chang, M., Liu, H. & Tai, C.Y. (2011). Powder Technology, 207, 378–386.
- Cruccolini, A., Narducci, R. & Palombari, R. (2004). Sensors Actuators B, 98, 227–232.
- Eastman, J.A, Choi, S.U.S, Li, S. Yu, W. & Thompson, L.J. (2001). Anomalous increased effective thermal conductivities of ethylene glycol based nanofluids containing copper nanoparticles. Applied Physics Letter, 78, 718–720.
- Krithiga, N., Jayachitra, A. & Rajalakshmi, A. (2013). Synthesis, characterization and analysis of the effect of copper oxide nanoparticles in biological systems. Indian journal of Nanoscience, 1, 6–15.
- Kumar, A.S., Meenakshi, K.S., Narashimhan, B.R.V, Srikanth, S. & Arthanareeswaran G. (2009). Synthesis and characterization of copper nanofluid by a novel one step method. Material Chemistry and Physics, 113, 57–62.
- Li, Y., Liang, Z. Tao & J. Chen. (2008). Material Research Bulletin, 43, 2380–2385.
- Liu, M.S, Lin, M.C.C, Tsai, C.Y. & Wang, C.C. (2006). Enhancement of thermal conductivity with Cu for nanofluids using chemical reduction method. International Journal of Heat and Mass Transfer, 49, 3028–3033.
- Mallinson, J.C. (1987). The Foundations of Magnetic Recording. Chapter 3. Academic Press, Berkeley, CA.
- Mitsuyu, T., Yamakazi, O., Ohji, K. & Wasa, K. (1982). Ferroelectrics, 42 233. 119
- Rakhshni, A.E. (1986). Solid State Electron, 29, 7.
- Sambandam Anandan & Shine Yang, (2007). “Emergent Methods to Synthesize and Characterize Semiconductor CuO nano particles with various morphologies – an overview. Journal of Experimental Nanoscience, 2, 1- 2, p.2356.

13. Toraya H. (1986). Whole- powder- pattern fitting without reference to structural model: application to x- ray powder diffraction data. *Journal of Applied Crystallography*, 19, 440-447.
14. Wang, H., Xu, J., Zhu, J. &Chen, H. (2002). *Journal of Crystal Growth*. 244: 88–94.
15. Wolhfarth, E.P. (1980). *Ferromagnetic Materials*, Vol. II, North-Holland, Amsterdam, New York, Oxford, Tokyo, 405.
16. Xuan, Y. & Li, Q. (2000) Heat transfer enhancement of nanofluids. *International Journal of Heat Fluid Flow*, 21, 58–64
17. Kim, Y.S., Hwang, I.S., Kim, S.J., Lee C.Y. & Lee, J.H. (2008). *Sensors Actuators B*, 135, 298– 303.
18. Zhang, J., Liu, Q., Peng, X., Wang, Y. & Li. (2006). *Chemistry of Materials*, 18, 867–871.